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WAL TR 766.2/3-3

FOURTH QUARTERLY PROGRESS REPORT
EVALUATION OF HIGH-STRENGTH LIGHTWEIGHT LAMINATED PRESSURE VESSELS OF LAP-JOINT CONSTRUCTION

by
G. Citrin

(RAC 1160, 244-3004)

16 January 1963

Period covered: 1 October to 31 December 1962
Republic Aviation Corporation
Mineola, L. I., N. Y.

Contract DA-30-069-ORD-3440
New York Procurement District, U. S. Army

00 Project No. -OMS Code 5010.11.8430051

Department of the Army Project No. 59332008
Watertown Arsenal
Watertown 72, Massachusetts

TECHNICAL REPORT NO. WAL TR 766.2/3-3

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ABSTRACT

Fourth Quarterly Progress Report
Evaluation of High-Strength, Lightweight
Laminated Pressure Vessels of
Lap-Joint Construction

During this quarter, the production sheet metal material has been delivered and evaluated against specification requirements. Rings were fabricated of the 0.021-inch thick mar-aging steel material for the first three pressure vessels. The first pressure vessel was assembled and tested successfully producing a burst strength 7.9 percent greater than that indicated by uniaxial tensile tests of the parent sheet metal.

SECTION I

INTRODUCTION

A. PURPOSE

This contract is being performed to demonstrate the basic feasibility and the achievable confidence level of the overlapping cylinder design as applied to the production of high-strength pressure vessels made of high-nickel, mar-aging steels. Three nominal thicknesses of material, 0.025, 0.040, and 0.064 inch, are being used for the evaluation of the overlapping cylinder design on pressure vessels having wall thicknesses of 0.050, 0.080, and 0.128 inch, respectively. The pressure vessels will be nominally 24 inches in diameter and from 40 to 90 inches long. Phases 1 and 2 are investigations of structural adhesives and of brazed or ceramic bonded joints, respectively. Republic is working only on Phase 1. Phase 2 work is being done at Watertown Arsenal Laboratories.

B. SUMMARY OF ACTIVITIES COVERED BY PREVIOUS REPORTS

The effort during the first quarter, which was primarily on the materials survey, resulted in a tentative selection of the 18-percent-nickel, mar-aging steels as having the greatest potential usefulness. Small quantities of sample materials were evaluated. The basic weldability characteristics were found to be excellent and the potential of these alloys for parent-metal operation at the 300,000-psi yield-strength levels was confirmed. Preliminary tests also indicated satisfactory bonding characteristics. The results of these probing tests were sufficiently encouraging to warrant ordering adequate test quantities of both the 18-7-5 (INCO 250 KSI) and 18-9-5 (INCO 300 KSI) NiCoMo, production-type alloys.

During the second quarter, the test quantities of material were received and evaluated sufficiently to ensure their suitability for this program. A final selection of the 18-9-5 (INCO 300 KSI) composition was made, and the pressure-vessel material was ordered.

During these two periods, the tool design and a major portion of the tool fabrication were completed. The lap-shear test program for adhesive selection

was started, and preliminary subscale ring-sizing tests were successfully performed using welded preforms made of the 18-9-5 NiCoMo alloy.

During the third quarter, the lap-shear testing program for the selection of adhesives was completed. The designs for the work-horse headers were also completed. Header-machining and heat-treat operations were started.

The remaining subscale ring-sizing tests were performed, and the necessary allowances for variables, such as spring back and subsequent aging shrinkage, were established to ensure dimensional control.

The first delivery of mar-aging steel for production was made in September and its evaluation was started.

C. SUMMARY OF ACTIVITIES COVERED BY THIS REPORT

The major efforts during this period were expended on resolving the mar-aging steel production problems at the mill and on fabricating and testing the first pressure vessel.

The first partial delivery of production material (received in September) did not comply with several of the specification requirements and, therefore, was sent back to the mill for reprocessing. Usable production materials but with reduced thicknesses were finally available in the middle of November, 1962.

In December, assembly of the first pressure vessel was started using 0.021-inch-thick steel (originally 0.025 inch thick) and polyamide-epoxy (FM 1000) adhesive. This vessel was hydrostatically tested; it burst at 990 psig internal pressure and withstood a hoop stress of 286,000 psi in the mar-aging steel wall. There was no failure or permanent deformation in the adhesive joint, and the cylinder weldments showed no primary failures.

D. SUMMARY OF ACTIVITIES PLANNED FOR NEXT QUARTER

During the next quarter additional pressure vessels will be built and tested. All three thicknesses of material will be used for these vessels. The optimum adhesive and vessel design will be established for each material thickness.

Process development work will be performed so that the various problems encountered during the assembly of the first pressure vessel can be eliminated.

SECTION II

MATERIALS USED

A. GENERAL

A complete evaluation of the initial shipment of production material was made. It was found that the solution-treatment mill cycle had been too short and had resulted in severe nonuniformity in the mechanical properties. The material was subsequently reprocessed at the mill and delivered to Republic in November. The corrective action proved to be effective in eliminating the nonuniformity. However, a reduction in thickness had to be authorized in order to make the material flat. The contractor found that a revision in aging temperature from 900 to 925 degrees F was required before satisfactory properties could be developed for this heat of material.

The lap-shear tests to select the adhesives were completed. The design parameters previously selected (by interpolation) for the 0.040-inch material were confirmed.

B. WELDING AND METALLURGICAL EVALUATION

Work during the fourth quarter included evaluation of previously determined and modified welding schedules on the original shipment of metal as well as on the reprocessed production material in 0.021 and 0.038 gages. Rings have been welded and sized in the 0.021 gage. Because of the poorer surface finish of this material than that of the test material previously used, a slightly concave weld (0.001 to 0.002 inch deep) resulted. During ring sizing runs, it was found that less deformation could be tolerated because of the slight reduction in thickness at the weld. It was necessary, therefore, to investigate supplementary weld filler wire on the 0.038- and 0.021-inch thick materials. This welding schedule revision results in a convexity totaling 0.004 inch maximum on the weld bead. Tests on the production heat further substantiate the results obtained previously, namely that weldability and the resulting weld characteristics of mar-aging steel sheet materials are substantially affected by the material condition (cold worked, aged, or annealed) prior to welding.

Data on the mechanical properties of the production material parent metal and weldments before and after reprocessing by the mill are presented in Tables 1 and 2.

The most significant tests are those made in the as-received condition and those made after solution treatment at 1500 degrees F for 15 minutes. It will be noted that the former resulted in relatively high, nonuniform strengths while the latter resulted in much lower, quite uniform strengths. This seemed conclusive evidence of inadequate solution treatment at the mill.

When the material was returned from the mill after reprocessing to correct the solution treatment inadequacy, it was found that its mechanical properties were somewhat lower than expected in the 0.021-inch-thick material after aging at 900 degrees F for 3 hours. Knowing that variations in optimum aging temperature occur as a function of very small chemistry variations, an investigation of both higher and lower aging temperatures was made. A very real improvement in both strength and elastic modulus was found when a 925-degree F aging temperature was used. This change will be incorporated in all parts manufacturing using this heat of material; subject to confirmation tests in the other two thicknesses.

Data on the effect of heat treat cycle on mechanical properties are included in Table 3 along with data of three control specimens processed along with the rings incorporated in assembly III-101.

C. FORMABILITY EVALUATION

Work was started on the fabrication of full-scale production rings. A total of 11 rings, 0.021 inch thick, was fabricated for the III-101, III-102, and V-103 assemblies. (Reference "Third Quarterly Report, RAC 954, subsection III, Figure 5 and Table 7, pages 24 and 25.)

The results of this work confirmed the data previously reported with regard to the dimensional control that is achieved by the sizing process and the dimensional stability of the 18-9-5 NiCoMo (300 ksi) mar-aging steel. Table 4 is a summary of the results of the ring fabrication for the first three 0.021 assemblies. The manufacturing procedure is basically as previously reported for the subscale ring sizing program ("Second Quarterly Report," MSD 244-3002, subsection II C, pages 20 to 24) except for the aging cycle, which is 925 degrees F for 3 hours.

TABLE 1. MECHANICAL PROPERTIES OF PRODUCTION MATERIAL AS ORIGINALLY RECEIVED

Specimen Condition	18-9-5 NiCoMo (300-KSI) Mar-Aging Steel Specimen Characteristics				
	Load Direction*	Nominal Thickness (inch)	0.2-Percent Offset Yield Strength (KSI)	Ultimate Tensile Strength (KSI)	Elongation in 2 Inches (percent)
As received	T	0.064	157.9	198.7	12.0
	L	0.064	134.1	175.5	8.5
	L	0.064	130.4	176.0	8.5
	L	0.064	131.9	175.7	8.5
	L	0.064	128.2	173.5	8.0
	T	0.040	134.3	185.5	7.5
	L	0.040	132.8	169.8	6.0
	L	0.025	115.0	158.6	5.5
Specification requirements	-	-	135.0 max	165.0 max	8.0 min
As received and re-solution treated	L	0.064	114.9	181.0	9.0
	L	0.064	118.7	179.7	9.0
	L	0.064	119.6	178.5	8.5
As received and aged at 900 degrees F for 3 hours	T	0.064	343.4	346.9	5.0
	L	0.064	312.5	312.5	2.0
	T	0.040	335.4	338.9	3.0
	L	0.040	--	302.6	2.0
	L	0.025	268.9	272.3	1.5
Specification requirements	-	-	270.0 min	280.0 min	3.0 min
As received, solution treated and welded	L	0.040	113.7	141.9	4.0
	L	0.040	122.3	146.7	3.5
As received, welded and solution treated	L	0.040	104.0	152.9	5.5
As received and welded	L	0.040	128.0	153.3	4.0
As received, solution treated, welded and solution treated	L	0.040	112.1	147.9	3.5
	L	0.040	113.8	147.2	3.5

* T is transverse; L is longitudinal; relative to the grain of the metal

TABLE 2. MECHANICAL PROPERTIES OF PRODUCTION MATERIAL REPROCESSED BY MILL

Specimen Condition	18-9-5 NiCoMo (300-KSI) Mar-Aging Steel Specimen Characteristics				
	Load Direction*	Nominal Thickness (inch)	0.2-Percent Offset Yield Strength (KSI)	Ultimate Tensile Strength (KSI)	Elongation in 2 Inches (percent)
As received	T	0.021	103.9	144.8	3.0
	T	0.021	102.6	141.7	4.5
	T	0.021	106.8	128.2	3.5
	T	0.021	106.8	128.2	3.5
	L	0.021	102.0	139.3	5.0
	L	0.021	97.4	134.2	5.0
	L	0.021	105.8	122.8	4.0
	L	0.021	92.2	123.6	3.5
	T	0.038	124.5	147.4	5.0
	T	0.038	125.4	148.7	5.5
	L	0.038	119.7	142.0	6.0
	L	0.038	113.0	139.9	5.5
	L	0.038	114.1	140.1	6.0
	T	0.062	126.8	154.2	6.5
	T	0.062	126.3	154.5	6.5
	L	0.062	118.8	146.3	6.5
	L	0.062	119.3	146.8	7.0
	L	0.062	121.2	148.1	6.5
As received and aged for 3 hours at 900 degrees F	T	0.021	231.9	237.1	2.0
	T	0.021	235.6	240.4	2.5
	T	0.021	236.9	241.7	3.0
	L	0.021	223.3	225.2	2.0
	L	0.021	229.2	234.2	2.0
	L	0.021	224.0	228.1	2.0
	T	0.038	276.7	282.4	3.0

* T is transverse; L is longitudinal, relative to the grain of the metal

TABLE 2. MECHANICAL PROPERTIES OF PRODUCTION MATERIAL REPROCESSED BY MILL (cont'd)

Specimen Condition	18-9-5 NiCoMo (300-KSI) Mar-Aging Steel Specimen Characteristics				
	Load Direction*	Nominal Thickness (inch)	0.2-Percent Offset Yield Strength (KSI)	Ultimate Tensile Strength (KSI)	Elongation in 2 Inches (percent)
As received and aged for 3 hours at 900 degrees F (cont'd)	T	0.038	274.9	280.0	3.0
	T	0.038	276.4	281.0	3.0
	L	0.038	266.8	268.4	3.0
	L	0.038	265.8	268.4	2.0
	L	0.038	260.8	263.5	2.0
	T	0.062	283.5	288.8	3.5
	T	0.062	279.9	287.3	3.5
	T	0.062	282.3	288.8	3.5
	L	0.062	272.5	275.3	4.0
	L	0.062	271.5	274.7	4.0
	L	0.062	272.6	275.8	3.5
As received and welded	L	0.021	101.5	132.0	2.5
	L	0.021	103.3	131.9	3.0
	L	0.062	108.8	143.9	4.0
	L	0.062	114.9	149.7	4.0
	L	0.062	109.7	144.0	4.5
	L	0.062	113.7	149.0	4.5
As received, welded and aged at 925 degrees F for 3 hours	L	0.021	238.2	243.2	0.0
	L	0.021	238.1	243.2	0.5
	L	0.062	261.9	265.2	1.5
	L	0.062	260.4	263.0	1.5
	L	0.062	261.3	264.8	1.5
	L	0.062	260.4	263.0	2.0

* T is transverse; L is longitudinal; relative to the grain of the metal

TABLE 3. MECHANICAL PROPERTIES AS A FUNCTION OF AGING TEMPERATURE

Specimen Condition		18-9-5 NiCoMo (300 KSI) Mar-Aging Steel Specimen Characteristics				
Aging Temperature (degrees F)	Time (hours)	Load Direction*	Nominal Thickness (inch)	0.2-Percent Offset Yield Strength (KSI)	Ultimate Tensile Strength (KSI)	Elongation in 2 Inches (percent)
875	3	L	0.021	241.4	256.0	2.5
875	3	L	0.021	246.6	257.8	2.5
875	3	L	0.021	232.5	251.3	3.0
900	3	L	0.021	237.8	250.9	5.0
900	3	L	0.021	245.6	251.3	5.0
900	3	L	0.021	250.4	257.0	5.0
900	3	T	0.021	229.1	242.3	5.0
900	3	T	0.021	241.8	255.5	5.0
900	3	T	0.021	243.5	255.1	5.0
925**	3	L	0.021	250.0	266.4	1.5
925**	3	L	0.021	238.2	265.5	2.0
925**	3	L	0.021	245.7	260.8	1.5
925	3	T	0.021	264.7	281.4	1.5
925	3	T	0.021	264.3	280.5	1.5
925	3	T	0.021	269.5	280.0	1.0
925***	3	T	0.021	266.4	280.4	3.0
925***	3	T	0.021	260.5	276.7	2.5
925***	3	T	0.021	257.1	272.4	1.5

* T is transverse; L is longitudinal; relative to the grain of the metal

** The average longitudinal strength of the 0.021 III-101 vessel is 264.2 ksi

*** Control specimens accompanying rings used in assembly III-101 during heat treatment

TABLE 4. SUMMARY OF RESULTS FOR 0.021-INCH-THICK RING FABRICATION

Assembly Number	Part Number*	Preform Diameter** (inches)	Total Deformation (percent)	Total Permanent Set (percent)	Diameter before Aging** (inches)	Shrinkage in Aging (inch)	Diameter after Aging** (inches)
III-101	2-6-2-A	23.569 O	1.62	0.75	23.738 O	0.017	23.721 O
	2-6-9-A	23.540 O	1.62	0.81	23.728 O	0.018	23.710 O
	2-6-10-B	23.536 I	1.62	0.87	23.734 I	0.017	23.717 I
III-102	2-10-5-A	23.610 O	0.94	0.31	23.685 O	0.019	23.666 O
	2-10-4-A	23.616 O	0.94	0.31	23.688 O	0.020	23.668 O
	2-8-2-B	23.572 I	1.38	0.62	23.720 I	0.018	23.702 I
V-103	2-6-1-B	23.513 I	1.62	0.87	23.720 I	0.018	23.702 I
	2-8-6-A	23.585 O	1.38	0.56	23.716 O	0.020	23.696 O
	2-6-3-B	23.525 I	1.62	0.81	23.718 I	0.018	23.700 I
	2-10-2-A	23.625 O	1.00	0.44	23.717 O	0.017	23.700 O
	2-10-3-A	23.620 O	1.00	0.44	23.717 O	0.017	23.700 O

* A = inner ring
B = outer ring

** I = inside diameter
O = outside diameter

As shown in Table 1, production material that was delivered first had some discrepancies with respect to an overstrength condition; Specification MSD 244-4201 calls for a yield strength of 135 ksi maximum in the as-received condition. Tests showed yield strengths as high as 157.9 ksi. The weldability was satisfactory, but, when test cylinders were sized, they broke prematurely in the weld. The ductility evidenced was great, but because of a combination of a nonuniform, high yield strength in the parent metal and the reduced strength in the weld, all of the deformation occurred in the weld.

Tensile tests of as-received specimens and of as-received and re-solution treated specimens in Table 1 illustrate that the high yield strength condition could be remedied by a full re-solution treatment, which was accomplished at the mill.

D. SURFACE PREPARATION/BONDING EVALUATION

The lap shear test program was essentially completed during the fourth quarter. Only "Test Series E, Cyclic Testing" remains to be performed. The cyclic testing of the lap shear specimens (Load specimen to proof level, hold load for various periods, drop load to zero and recycle two more times, finally raise load till specimen fails.) to determine joint behavior under simulated acceptance test procedures will be delayed until data is available on the proof loading to be used for pressure vessel evaluation based on burst data for the initial pressure vessels of each thickness. The confirmation tests on the 0.040-inch-thick production material have been performed and the joint design criteria, which were interpolated and presented in the third quarterly progress report (Table 6), have been substantiated. The adhesives chosen for use with the 0.040-inch-thick material are AF-41 and FM1000. The results of these tests appear in Tables 5 and 6. The configuration of a typical lap shear specimen is shown in Figure 1.

TABLE 5. LAP SHEAR TEST C*

Lap Length (inches)	Number of Specimens	Adhesive	Ultimate Breaking Load (pounds)
6	3	FM 47	11,500, 12,500, 12,750
6	3	FM 1000	12,800, 13,150, 13,700
6	3	AF 41	12,300, 12,400, 12,700
7	3	FM 47	10,500, 11,400, 12,100
7	3	FM 1000	13,700, 13,700, 13,750
7	3	AF 41	12,000, 12,800, 12,900

* All specimens 0.040 inch thick adherents.
All surface preparation for the adherents consisted of vapor degrease followed by fine-grit liquid hone.

TABLE 6. LAP SHEAR TEST D*

Lap Length (inches)	Number of Specimens	Adhesive	Ultimate Breaking Load (pounds)
7	5	FM 47	9000, 10,000, 10,400, 11,000, 12,100
7	5	FM 1000	12,000, 12,100, 12,300, 12,500, 12,650
7	5	AF 41	12,000, 12,200, 12,250, 12,250, 12,700

* Configuration of lap shear specimens were 0.040 inch gage 18-9-5 NiCoMo on 0.090-inch D6-A steel. The surface preparation process for the 0.040 inch 18-9-5 NiCoMo was vapor degrease followed by fine grit liquid hone; vapor degrease followed by dry, coarse-grit blast was used for the surface of the D6-A steel.

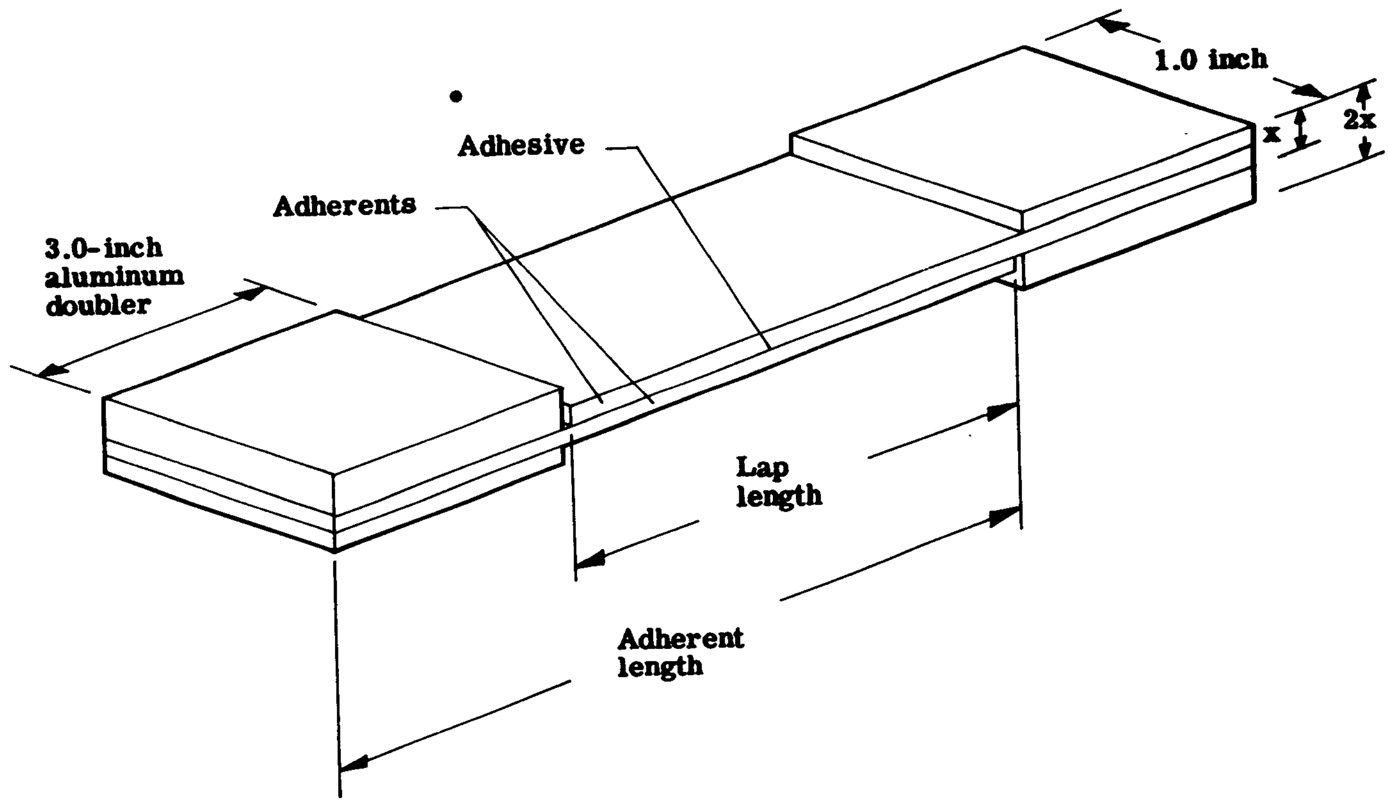

Figure 1. Typical Lap-Shear-Specimen Geometry

SECTION III

DESIGN AND TEST

During the fourth quarter, pressure vessel assembly III-101 was hydrotested to failure (Figure 2). The vessel tested departed from the normal configuration, as shown in Figure 5 of "Third Quarterly Progress Report," RAC number 954. It was necessary to add a 17-7 PH steel hoop (1.00 by 0.064 inch) to the assembly because a 1-inch longitudinal gap occurred during the final assembly operation (see Section IV).

The vessel was pressurized to 205 psi for 5 minutes in order to check the setup for leaks. The pressure was then increased uniformly for 67 seconds until failure occurred at 990 psi internal pressure. The corresponding membrane hoop stress in the vessel at failure was 286,000 psi, exceeding by 7.9 percent the uniaxial tensile strength of the material used (see Table 3).

A permanent record of the pressure and time history of the test was made using a C. E. C. oscillograph. Dial gages were used during the test for visual control of the test. Prior to the test all of the instruments were calibrated using standard procedures.

Although the failure was extremely difficult to analyze and a distinct failure origin (cleavage) was not observed, it is believed that the primary failure originated in the cylindrical section of the motor case assembly near the end of the dome straight section.

Figures 2 and 3 are photographs of the case after failure. A line drawing of the probable crack propagation is shown in Figure 4. An examination at a magnification of 45 times with a wide-field stereomicroscope revealed a fracture pattern similar to that seen in monolithic construction. Photographs of the axial fracture propagation (Figures 5 and 6) show that the fracture surfaces are almost entirely sheared. Only those areas that were subjected to large bending stresses late in the failure showed cleavage-like indications. Figure 7 shows a typical fracture surface.

Figure 2. View of Primary Failure Zone of Assembly III-101

MR-3016

Figure 3. View of Assembly Components after Test

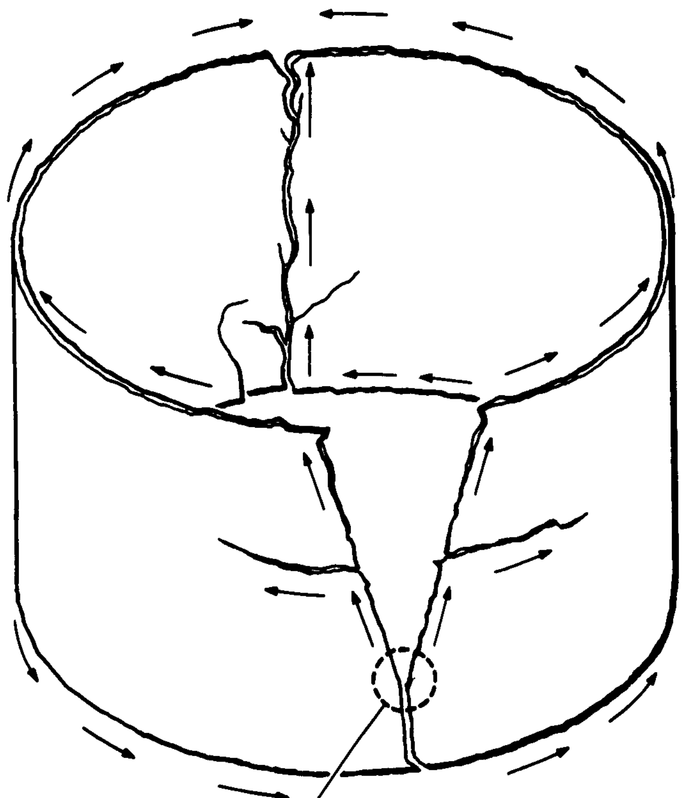

Figure 4. Probable Crack Propagation Path

MR-3008

Figure 5. Close-up of Secondary Failure Area

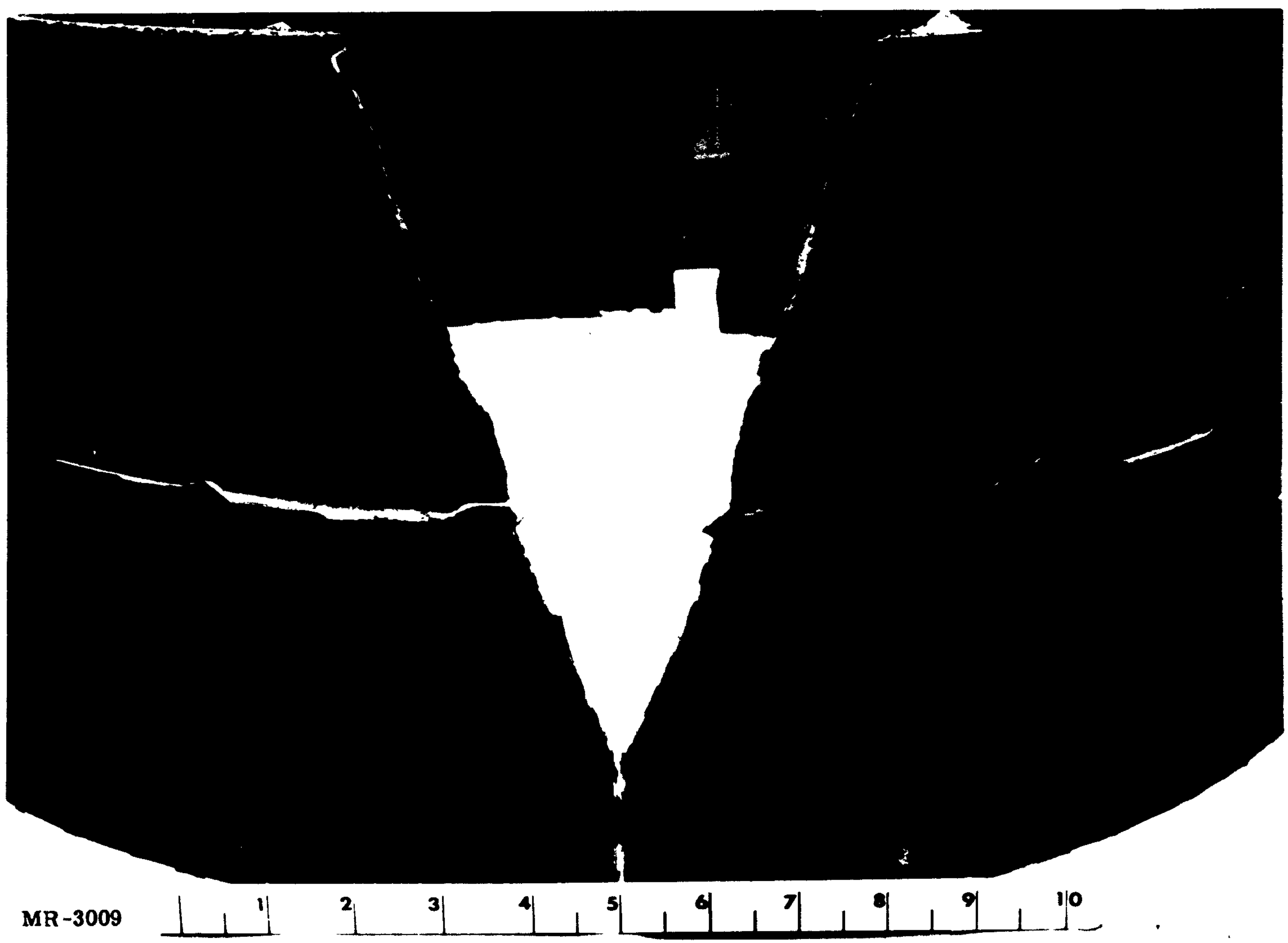

Figure 6. Close-up of Primary Failure Area

MR-3010

Figure 7. Typical Fracture Surface
Magnified About 5 Times

SECTION IV

TOOLING AND MANUFACTURING RESULTS

The assembly process consists of heating outside rings and headers by means of heating blankets thus increasing their diameter. This permits their assembly over the inner rings on which the adhesive has been positioned. The resulting assembly is then cured in an oven according to the requirement of the adhesive used. Prior to testing, a sealant is applied to the interior of the vessel.

The heating blankets were received during this quarter and their operation at temperatures to 500 degrees F was verified.

During the assembly of pressure vessel III-101, the operational characteristics of the bonding assembly fixture were determined to be marginal. The difficulties encountered, mainly lack of alignment and speed, caused the header to bind 1.00 inch before reaching the proper position. Therefore, a hoop ring was added to prevent premature failure during the hydrotest (see Section III). This was justified by the fact that all header joints have been designed 1 inch longer than the body joints and the mar-aging steel to D6-A steel lap shear test joint strengths were all equal to or higher than the equivalent lap length of mar-aging to mar-aging steel lap shear tests. The difficulties are being analyzed and will be solved during the next quarter.

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